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Preparation and Characterisation of Ni(II), Pd(II) and Pt(II) Complexes of 1-Diphenylphosphino-2-bis(*m*-trifluoromethylphenyl)phosphinoethane, (*m*-CF_3 P–P)

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Abstract

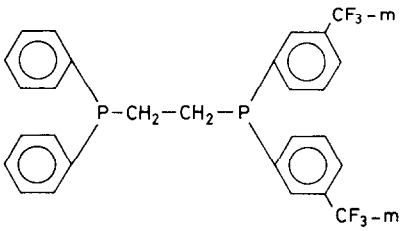
A series of four-coordinate, square-planar, diamagnetic 1-diphenylphosphino-2-bis(*m*-trifluoromethylphenyl)phosphinoethane complexes of type *cis*-[MX_2(*m*-CF_3P–P)] (M = Ni, Pd, Pt; X = Cl, Br, I or NCS) have been prepared. These complexes have been characterized by $^{31}P\{^1H\}$ NMR, ^1H NMR, IR and UV spectroscopy, elemental analyses and magnetic susceptibility measurements. The effects of various substituents on the phenyl groups of the ditertiary phosphines on the solubility characteristics of the metal complexes are discussed.

Introduction

The coordination chemistry of the symmetrical ditertiary phosphines, $R_2PCH_2CH_2PR_2$, is well documented and is dominated by the formation of stable five-membered ring chelate complexes [1–6]. Ten-membered ring binuclear complexes have also been reported recently [7]. In a few cases, these also behave as monodentate or bridging ligands, and have been used in the synthesis of heterobimetallic complexes [8–10].

In contrast to the above, few metal complexes of unsymmetrical ditertiary phosphines have been reported [11–14]. Studies of these unsymmetrical ditertiary phosphines are of considerable interest, since they contain in the same molecule, two phosphorus atoms which are magnetically non-equivalent, thus demonstrating the potential of ^{31}P NMR studies as a probe of stereochemical behaviour of the resulting transition-metal complexes [14–16].

Although the ditertiary phosphine ($Ph_2PCH_2CH_2PPh_2$) complexes of Ni(II), Pd(II) and Pt(II) were reported about two decades ago [17, 18], few attempts have been made [12, 19, 20] to study unsymmetrical ditertiary phosphines containing electronegative substituents on the phosphorus atoms. In an earlier publication [14], we reported the Ni(II), Pd(II) and Pt(II) complexes of two unsymmetrical ditertiary phosphines (*p*- or *m*-$FC_6H_4)_2PCH_2CH_2PPh_2$. This paper describes an analogous series of Ni, Pd and Pt complexes of the ditertiary phosphine ligand (*m*-CF_3P–P), the synthesis of which was reported earlier [21].



(m-CF_3P–P)

Experimental

Physical measurements and experimental techniques were carried out as described elsewhere [14].

Synthesis of Complexes

1. [Ni(m-CF_3P–P)Cl_2]

$NiCl_2 \cdot 6H_2O$ (0.19 g, 0.78 mmol) dissolved in butan-1-ol (30 ml) was refluxed for 1 h to produce a light green solution. To this, 1-diphenylphosphino-2-bis(*m*-trifluoromethylphenyl)phosphinoethane (0.42 g, 0.78 mmol) was added, giving a deep red solution. The reaction mixture was then refluxed for 2 h. On cooling, red crystals were deposited which were collected on a sintered glass crucible and crystallized from a mixture of methylene chloride and n-hexane.

2. [Ni(m-CF_3P–P)Br_2]

This complex was prepared as described above, $NiBr_2$ being used. Recrystallization of the crude

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